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#### A METHOD FOR GRANULATION OF A CERAMIC POWDER BODY

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# (54) A METHOD FOR GRANULATION OF A CERAMIC POWDER BODY

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(56) References:

Japanese Patent Sho 36(1961)-15264 (JP, Bl) Japanese Patent Sho 36(1961)-15265 (JP, Bl)

### (57) Scope of Patent Claim

A method for granulation of a ceramic powder body which
is characterized by the fact that it consists of the process
whereby a glue that has been predissolved in a solvent and
a ceramic powder are mixed, the process whereby the solvent is
removed by sufficient drying of this mixture, the process
whereby the mixture is then pulverized further, the process
whereby a volatile liquid in which the aforementioned glue is insoluble
is added and mixed to make a cloudy substance, and the
process whereby this cloudy substance is granulated.

Detailed Explanation of Invention

This invention pertains to a method for granulation of a ceramic powder body suitable for pressed powder and molds.

Heretofore, the method whereby a glue that has been predissolved in a solvent is added to a ceramic powder and this mixture is then mixed with a mixer, kneader, etc., granulated and dried, or the method whereby a pulverized and granulated substance is placed in a mold and molded have been used as method for making ceramic molds.

However, the ceramic powder obtained with these methods becomes very hard during drying because of the glue that is added. Therefore, spaces between the granules easily form during molding and therefore, a higher than normal molding pressure is necessary. This hardening effect becomes worse

<sup>\*</sup> Numbers in margin indicate foreign pagination

with more effective glues. In order to solve this problem, the method whereby dried powder is further granulated has been used. However, in this case the filling ability is poor and becomes worse with a granulated starting powder. Moreover, this phenomenon is particularly bad when water is used as the solvent. Therefore, there is a problem with methods employing the aforementioned granulation process from a practical standpoint since water is preferred as a solvent. For instance, when flaws are produced between the granules in rubber press molds, where a vertical filling ability is necessary, the final product will leak.

This invention improves on the aforementioned points. Its
purpose is to present a ceramic powder granule which has good filling
ability and with which there is little chance of cracks being produced
between granules during molding. That is, this invention is
a method for granulation of ceramic powder bodies which
is characterized by the fact that after a glue that has been
predissolved in a solvent and a ceramic powder are mixed together,
sufficiently dried, the solvent is removed and the mixture is
further pulverized. Then a nonvolatile liquid in which the aforementioned
glue is insoluble is added and mixed to make a cloudy
substance and this cloudy substance is then granulated with
conventional methods.

The inventors of this invention discovered that a ceramic granulated powder with superior filling ability and which did not produce cracks between the granules during molding could be obtained due to the plasticity provided during granulation by granulating the powder itself into particles of a suitable size, because the glue itself is evenly mixed with the starting powder.

Consequently, it is very important that the glue be uniformly mixed with the starting powder after it has been dissolved in a solvent and that after the solvent is vaporized, the mixture be further granulated with a suitable liquid in which the glue is insoluble. The granulation process in this invention can be carried out with any suitable conventional method, such as the method whereby mixers are employed. Example 1

30 wt of a 5% aqueous solution of polyvinyl alcohol was added to alumina powder with a mean particle diameter of 1 micron or less. After this was mixed with a mixer, it was sufficiently dried with the air bus method to obtain an alumina powder mass with a uniform 1.5 wt% polyvinyl alcohol content.

Part of this mass was granulated with conventional methods of by being passed through a 4 mesh sieve. The other part was placed in a mixer once more and made into coarse granules. Then methyl alcohol was added and the granules were pulverized. After they were small enough, they were semidried. Then these particles were passed through a 40 mesh sieve to obtain the granulated powder in this invention.

These granules were molded into pellets under a pressure of 1 t/cm<sup>2</sup> using a 20 \$\phi\$ mold. The mold density of the pellets was then measured. Next, a sintered body was obtained by baking these pellets in air at 1600 °C for 1 hour. The density and compressive strength of the sintered body was measured. The effects of the method in this invention and conventional methods on finished goods were compared.

The aforementioned pellet molds were also split into 2 pieces and observed with an SEM. 95% of the pellet obtained

with conventional methods crumiled. Moreover, air holes were seen between the granules. In constrast to this, only 45% of the pellet obtained with the method in this invention crumbled from the outside and 65% of the inside of the pellet crumbled. Air holes were not seen. Since there were no air holes between granules, the granulated powder obtained with conventional methods was pulverized to 100 mesh or less. This was then molded as previously mentioned and dried. Its properties were then studied. Moreover, three types of of powders were filled into a 5 \$ x 300 l rubber mold. The time it took to fill the mold was measured and the filling manageability was compared.

The following table shows the results of the studies.

	Conventional method		This invention
	40 mesh	100 mesh	40 mesh
molding density	$1.8 \text{ g/cm}^3$	$2.05 \text{ g/cm}^3$	$2.1 \text{ g/cm}^3$
sinter density	$3.7 \text{ g/cm}^3$	3.86 g/cm <sup>3</sup>	$3.92 \text{ g/cm}^3$
compressive stren	gth 25 tons/cm <sup>2</sup>	32 tons/cm <sup>2</sup>	36 tons cm <sup>2</sup>
filling time	4.5 sec.	52 seconds	3.2 seconds

As can be seen from the table, the properties of the conventional product were improved by granulation. However, there was a marked reduction in filling manageability.

The granulated powder from this invention displays good sinter properties and good filling manageability.

## Example 2

A  $10 \phi \times 1$  t  $\times 100$  1 tube was made with the method in Example 1 using light permeable alumina. The direct ray transmission factor was compared.

conventional this method

65%

direct ray transmission factor 5%

As can be seen from the table, the difference in the transmission factor with the conventional method and that with this invention is due to the fact that light is dispersed from air holes in the sinter formed during the molding period. This type of phenomenon was not seen with the granules obtained with this invention.